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# NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

TECHNICAL NOTE 2180

## EFFECTIVENESS OF MOLYBDENUM DISULFIDE AS A FRETTING-CORROSION INHIBITOR

By Douglas Godfrey and Edmond E. Bisson

Lewis Flight Propulsion Laboratory  
Cleveland, Ohio



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## SUMMARY

The effectiveness of molybdenum disulfide  $\text{MoS}_2$  as a fretting-corrosion inhibitor was investigated. Six methods of applying  $\text{MoS}_2$  to steel specimens were evaluated by noting the number of cycles of vibration required to produce the first evidence of fretting corrosion and by observing the nature of its occurrence.

Experiments were conducted with steel balls vibrating in contact with glass flats whereby the action could be microscopically observed. A coating of dry  $\text{MoS}_2$  (bonded to steel by rubbing a mixture of  $\text{MoS}_2$  and syrup into intimate contact with the clean steel at elevated temperatures) proved most effective. Of the methods investigated, this coating delayed the start of fretting corrosion of the steel ball to 28,000,000 cycles in contrast to less than 30 cycles for the clean uncoated steel ball. Dry  $\text{MoS}_2$  and mixtures of  $\text{MoS}_2$  with various carriers (water and aerosol, light oil, and heavy grease) appreciably delayed the occurrence of fretting corrosion. Microscopic observation, however, indicated that  $\text{MoS}_2$  may have been ineffective at the contact area and that the carrier alone may have been responsible for the beneficial action.

Experiments were also conducted with steel flats vibrating in contact with steel flats that produced fretting corrosion of clean untreated specimens in less than 100 cycles. A bonded coating of  $\text{MoS}_2$  was again most effective, delaying the start of fretting corrosion to 10,000,000 cycles, whereas dry  $\text{MoS}_2$  and mixtures of  $\text{MoS}_2$  with carriers were less effective. Observations indicated that the effectiveness of any fretting-corrosion inhibitor is dependent on its ability to prevent metallic adhesion continuously.

## INTRODUCTION

Fretting corrosion, defined as surface failure that may occur when closely fitting metal surfaces experience slight relative motion, damages many machine parts subject to vibration. Numerous occurrences of this phenomenon and resultant machine failures (described in reference 1) indicate that an urgent problem exists.

A great deal of empirical work has been done with inhibitors (for example, extreme-pressure oils and greases; oxygen-free lubricants; tin, copper, and chromium plating; and graphite); none of these inhibitors have appreciably minimized or prevented fretting corrosion.

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Recent research (reference 2) and earlier research (references 3 and 4) indicate that, basically, fretting corrosion is the result of localized or very concentrated friction phenomena. Based on the results of reference 2, adhesion is believed to be the primary friction phenomenon involved; adhesion causes the removal of finely divided oxidizable metal. Other phenomena, such as the rubbing off of oxide films, welding from frictional heat, and abrasion, undoubtedly contribute to further surface destruction.

Molybdenum disulfide  $\text{MoS}_2$  is a solid-film lubricant possessing the following desirable properties: high load-carrying capacity and capability of minimizing metallic adhesion (reference 5); tenacity for steel and low coefficient of friction (reference 6); and thermal stability (reference 7).

The compound  $\text{MoS}_2$  was therefore chosen as the most promising fretting-corrosion inhibitor of practical significance and an investigation was conducted at the NACA Lewis laboratory to determine its effectiveness. Experiments were conducted with steel balls vibrating in contact with glass flats at 120 cycles per second, an amplitude of 0.001 inch, and a normal load of 0.2 pound. Additional experiments were conducted with steel flats vibrating in contact with steel flats at 15 cycles per second, an amplitude of approximately 0.003 inch, and a nominal pressure of 20 pounds per square inch. Six simple and practical methods of applying  $\text{MoS}_2$  were used.

#### APPARATUS

Steel ball against glass flat. - Part of the experiments were conducted with essentially the same apparatus as that described in reference 2 by which fretting corrosion suitable for microscopic study was produced. A 1/2-inch-diameter chrome-alloy steel (SAE 52100) ball (surface finish, 3 to 4 microin. rms) was vibrated in contact with a clean glass microscope slide at a frequency of 120 cycles per second, an amplitude of 0.001 inch, and a load of 0.2 pound (calculated Hertz surface stress of 35,000 lb/sq in.). A diagrammatic sketch of the apparatus is presented in figure 1(a).

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Steel flat against steel flat. - In addition to the method of microscopic observation using steel vibrating in contact with glass, experiments were conducted with steel vibrating in contact with steel for better simulation of actual cases. A simple apparatus was used in which one clean flat specimen of blue Swedish spring steel was vibrated in contact with another at a frequency of 15 cycles per second, an amplitude of approximately 0.003 inch, and a nominal pressure of 20 pounds per square inch. A diagrammatic sketch of this apparatus is shown in figure 1(b). Swedish spring steel was chosen because, in practical use, this material is susceptible to failures by fretting corrosion.

#### PROCEDURE

The effectiveness of a number of methods of applying  $\text{MoS}_2$  was evaluated with both types of apparatus. The number of cycles to failure was determined in both cases by noting the elapsed time and by calculating these cycles from the theoretical frequency. Microscopic observation through the glass slide was made continuously for 100,000 cycles and periodically thereafter. In the experiments with steel flats against steel flats, the surfaces were separated and macroscopically examined every 5 minutes for 1 hour and every hour thereafter.

In some instances, fretting-corrosion inhibitors have been evaluated by noting specimen weight loss. When fretting corrosion occurs dry or in the presence of a liquid, this method is applicable if the stain and the debris are completely removed by the liquid or subsequent treatment. If the liquids or treatment only partly clean the specimen, the surface fretting may be more extensive than indicated by weight loss; if the surfaces are fretted dry, a gain in weight may result because of oxide formation. Fretting corrosion starts with sliding and material is removed from the surfaces during 1/2 cycle of vibration (reference 2). Because part of the removed material adheres tenaciously to the specimens, the weight-loss method may be incapable of detecting the inception of fretting corrosion. In this investigation, the very first appearance of debris or brown stain (indicating the presence of oxides) was taken as evidence of fretting corrosion.

All specimens were cleaned prior to application of  $\text{MoS}_2$  by scrubbing them in a commercial degreaser and by three successive washes in an uncontaminated solvent consisting of equal parts by

volume of acetone and benzene. As a basis of comparison, specimens cleaned according to this procedure (without any application of MoS<sub>2</sub>) were subjected to fretting corrosion. The six methods of applying the MoS<sub>2</sub> (commercial grade, 97.25 percent pure) to the steel balls and the steel flats were as follows:

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Method	Methods of application of MoS <sub>2</sub> to specimens
1	Dusted with MoS <sub>2</sub> powder
2	Rubbed with MoS <sub>2</sub> powder by fingers
3	Immersed in mixture of MoS <sub>2</sub> , distilled water, and aerosol
4	Immersed in mixture of MoS <sub>2</sub> and light oil (SAE 10)
5	Coated with mixture of MoS <sub>2</sub> and heavy grease (lime-soap base)
6	Bonded with MoS <sub>2</sub> by rubbing mixture of MoS <sub>2</sub> and corn syrup into intimate contact with cleaned hot metal

Photomicrographs of several treated steel surfaces as well as a cleaned surface are shown in part (a) of figures 2 to 5.

The coating of method 6 was formed by a procedure that is based on reference 8. The tenacity of the coating applied by this procedure may result from presence of an iron oxide; if so, the procedure could be applicable only to ferrous alloys. The procedure is as follows:

- (1) Thoroughly wash the specimen in a commercial solvent.
- (2) Completely degrease specimen by successive washes in uncontaminated solvent, such as equal parts by volume of acetone and benzene, until water will completely wet the surface.
- (3) Heat specimen until oxide film is dark blue.
- (4) Under a fume hood, dip and stir the hot specimen in a well-stirred mixture (almost boiling) of equal parts by volume of corn syrup and MoS<sub>2</sub> powder. For specimens with only certain areas to be coated, simply smear the hot mixture on those areas.

(5) Remove from mixture and while baking ( $250^{\circ}$  to  $600^{\circ}\text{F}$ ) until dry, thoroughly rub the specimen with a spatula to insure intimate contact between the mixture and the specimen.

(6) Cool the heavily coated specimen and rub off the excess mass to produce a smooth, slippery, gray, and uniform coating.

For very close-fitting parts, the film can be rubbed down to a thickness of only 0.0001 inch. Experiments reported in reference 6 indicate that a treatment with syrup alone causes a very small reduction in coefficient of friction as compared to a treatment with the mixture.

#### RESULTS

The results of this fretting-corrosion investigation are summarized in table I. The degree to which each method of applying  $\text{MoS}_2$  inhibits fretting corrosion is indicated by the number of cycles required for the occurrence of the phenomenon. The number of cycles is an average of three experiments and the results are reproducible within  $\pm 3$  percent except for method 5, the results of which were reproducible within only  $\pm 10$  percent. Observation of the behavior of the  $\text{MoS}_2$  and the nature of occurrence of fretting corrosion between steel balls and glass flats and between steel flats and steel flats is also briefly indicated in table I. For comparison, information on clean specimens is included.

#### Steel Ball against Glass Flat

Microscopic observation of the action at the area of contact was made; photomicrographs of the results for several methods of application are shown in figures 3 to 5. In these photomicrographs, black  $\text{MoS}_2$  is identified by white particles because the laminae efficiently reflect light. Fretting corrosion was identified by the appearance of red oxide, brown stain, and black active spots; however, the red oxide photographs as a light gray, finely divided, textured material, the brown stain appears as a local darkening, and the black spots photograph as black spots.

Clean steel balls and glass produced visible fretting corrosion instantaneously (less than 30 cycles) as is discussed in detail in reference 2 and shown in figure 2(b). Later stages are shown in figures 2(c) and 2(d). Conditions were therefore such that fretting corrosion would occur immediately unless delayed or prevented by other factors.

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The influence of applying  $\text{MoS}_2$  by dusting the dry powder on the surface (method 1) is shown in figure 3. Initially,  $\text{MoS}_2$  was in the area of contact (fig. 3(a)). With vibration the  $\text{MoS}_2$  was smeared; continued vibration rubbed this smeared film away, thus permitting metal-to-glass contact and consequent fretting-corrosion damage. This damage was evident after approximately 72,000 cycles (fig. 3(b)).

In the second method of application,  $\text{MoS}_2$  was thoroughly rubbed on the steel ball to encourage any inherent attraction; the surface appearance was similar to that shown in figure 3(a). Apparently the rubbing served only to reduce the amount of  $\text{MoS}_2$  and to remove larger particles. Because the steel balls were smooth, there were few microscopic valleys in which the  $\text{MoS}_2$  particles could be readily lodged. During vibration the  $\text{MoS}_2$  was smeared at the area of contact but fretting corrosion started at 21,600 cycles, a few hundred cycles after the  $\text{MoS}_2$  was rubbed away, and progressed in an uninhibited manner. (The appearance was similar to that shown in fig. 3(b).)

The third method of application using a mixture of  $\text{MoS}_2$  with water and aerosol resulted in a large amount of  $\text{MoS}_2$  in the area of contact; however, 1 second of vibration removed most of the visible  $\text{MoS}_2$ . After removal from the area of contact, observation revealed that the visible  $\text{MoS}_2$  vibrated sympathetically in the surrounding liquid and remained outside the area of contact. Fretting corrosion was first observed after approximately 21,600 cycles by the appearance of brown stain and red debris, which was dispersed in the water.

The deposition of a mixture of  $\text{MoS}_2$  and light oil (SAE 10) between the steel ball and the glass slide (method 4) placed  $\text{MoS}_2$  in the area of contact, as shown in figure 4(a). After a few seconds of vibration, however, the visible  $\text{MoS}_2$  had again receded, leaving oil at the area of contact (fig. 4(b)). After 86,400 cycles, fretting corrosion became evident as the usual brown stain and dispersed debris (fig. 4(c)). Once started, fretting corrosion was uninhibited and continued to the advanced stage shown in figure 4(d). When the bulk liquid surrounding the area of contact was drawn off leaving  $\text{MoS}_2$  and an oil film, fretting corrosion occurred with 1500 cycles and progressed uninhibitedly.

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The application of a mixture of MoS<sub>2</sub> and heavy grease (method 5) also delayed fretting corrosion. The mixture of MoS<sub>2</sub> and grease was present at the area of contact and the appearance was similar to that shown in figure 4(a). The visible MoS<sub>2</sub> was, however, absent after 3600 cycles of vibration, the appearance being similar to that shown in figure 4(b). In the early stages, wear of the steel ball was evident but fretting corrosion did not occur as defined herein. Fretting corrosion was delayed for approximately 216,000 cycles and then occurred, progressed, and appeared in a similar manner to that observed in the experiments with a mixture of MoS<sub>2</sub> and light oil (figs. 4(c) and (d)).

The extent to which a coating of MoS<sub>2</sub> bonded to the steel ball (method 6) delayed fretting corrosion is shown in figure 5. The nature of the undisturbed surface, with many reflecting surfaces, is shown in figure 5(a) to be completely covered and protected. A few seconds vibration changed the surface at the area of contact very little, but continued vibration produced the spot shown in figure 5(b). The black material appeared to be only finely divided MoS<sub>2</sub> and carbon. The MoS<sub>2</sub> coating after approximately 500,000 cycles is shown in figure 5(c); no great change had occurred and there was no action suggesting steel was about to be exposed. After 28,000,000 cycles, further pulverization had occurred and fretting corrosion was first evidenced as a brown stain (fig. 5(d)). This photomicrograph was taken without the glass in place to show how little the ball and the coating were damaged after being subjected to such a great number of cycles. The coating, however, was sufficiently worn to expose the metal.

#### Steel Flat against Steel Flat

The results of the second part of the experiments in which two flat specimens of blue Swedish spring steel were vibrated in contact are also summarized in table I. The determination was first made that the conditions of the experiments would immediately (less than 100 cycles) produce fretting corrosion between clean specimens. The extent of fretting corrosion between clean specimens after 4500 cycles is shown in figure 6. The debris, which photographs light gray, is a red-rust color. The clean portion of the specimen that was not subject to vibration is shown on the right side of the macrograph.

An example of fretting corrosion in the presence of unbonded MoS<sub>2</sub> is shown in figure 7, where the red stain and the debris were

visible after approximately 1,620,000 cycles with a mixture of MoS<sub>2</sub> and heavy grease (method 5) between the surfaces.

The mixtures of MoS<sub>2</sub> with oil (method 4) or grease (method 5) appreciably delayed fretting corrosion; however, the mixtures were to some extent "worked out" from between the flat specimens and left the bearing areas unprotected and with no evidence of deposited MoS<sub>2</sub>. Intermediate examination revealed the bare metal spots to be the areas where fretting corrosion subsequently occurred. Periodic examination of the flat specimens probably increased the number of cycles required to produce fretting corrosion because in spite of precautions, removing and replacing the specimens possibly smeared the incipiently fretted spots with lubricant.

The bonding procedure of method 6 formed a fine-textured continuous coating of MoS<sub>2</sub> on the flat steel. Vibration of a clean uncoated specimen in contact with the bonded specimens produced a number of shiny bearing areas and MoS<sub>2</sub> was transferred to the clean mating surface. After fretting corrosion was first evidenced at approximately 10,000,000 cycles the coating was very thin in spots and a brown tint from the oxide powder was visible. In addition, the MoS<sub>2</sub> coating was powdered in certain areas.

#### DISCUSSION

The results of the research with both types of experimental apparatus showed the same trend. The number of cycles required to produce fretting corrosion for each method of application indicated that a coating of dry MoS<sub>2</sub> bonded to steel was far superior to MoS<sub>2</sub> applied by other methods in delaying fretting corrosion. The mixtures of MoS<sub>2</sub> with oil or grease, however, appreciably delayed occurrence of fretting corrosion; all methods were beneficial.

Microscopic observation of the action between steel and glass with mixtures of MoS<sub>2</sub> and various carriers (water, oil, or grease) as inhibitors revealed that the MoS<sub>2</sub> may be ineffective. If so, the results are essentially for the carrier alone. In all the experiments with liquids, a finite time was apparently required to force the liquids from the area of contact, thus permitting metal-to-glass contact of sufficient magnitude to produce visible fretting corrosion. Further observation revealed that the surface of the steel ball was polished (that is, the microscopic scratches were obliterated) in the presence of liquids and without the usual evidence of fretting corrosion.

except for a slight clouding of the liquid. This observation indicates that boundary lubrication exists and that once conditions are favorable to permit a relatively great amount of real area of contact, sufficient debris is produced and extruded in a manner that inhibits further entrance of oil into the bearing area. Thus, fretting corrosion once started becomes self-accelerating.

Observations indicated that the effectiveness of any fretting-corrosion inhibitor is dependent on its ability to prevent metal-to-metal contact of surface asperities continuously. The circumstances under which fretting corrosion occurs are very demanding on the load-carrying capacity and continuity of any solid-film lubricant. The ploughing action of any one continuously reciprocating asperity would soon break through a film of low load-carrying capacity and after breaking up such a film (if it is nonregenerative) would proceed to push it out of the way.

The compound  $\text{MoS}_2$  is effective in preventing fretting corrosion as long as it remains in the areas of contact where it can minimize adhesion by preventing metal-to-metal contact of surface asperities. Properly bonded  $\text{MoS}_2$  appears capable of resisting the severe "cleaning" action of fretting corrosion for a long period of time. Some of the  $\text{MoS}_2$  is initially transferred to and lodged on the mating surface and thus covers the destructive asperities, bears part of the load, and facilitates the sliding. This mechanism is similar to that proposed in the theory of thin-metallic-film lubrication (reference 9).

#### SUMMARY OF RESULTS

The effectiveness of molybdenum disulfide  $\text{MoS}_2$  as a fretting-corrosion inhibitor was investigated. Six methods of applying  $\text{MoS}_2$  were used in the experiments with steel balls against glass flats and the action was microscopically observed. The more promising of these methods were used in experiments with steel flats vibrating in contact with steel flats and the specimens were periodically separated and examined. In both cases, the methods were evaluated by noting the number of cycles of vibration that produced the first evidence of fretting corrosion. The following results were observed:

1. A coating of dry  $\text{MoS}_2$  bonded to steel (by rubbing a mixture of  $\text{MoS}_2$  and syrup into intimate contact with the clean hot metal) proved most effective by delaying the start of fretting

corrosion to 28,000,000 cycles for steel balls against glass flats and approximately 10,000,000 cycles for steel flats against steel flats. Under the same conditions, clean uncoated steel specimens showed immediate (less than 100 cycles) fretting corrosion.

2. Dry MoS<sub>2</sub> and mixtures of MoS<sub>2</sub> with various carriers (water and aerosol, light oil, or heavy grease) appreciably delayed fretting corrosion (to as much as 216,000 cycles for the mixture of MoS<sub>2</sub> and heavy grease); however, microscopic observation revealed that MoS<sub>2</sub> may have been ineffective at the contact area. In the mixtures, it is possible that the carrier alone was responsible for the beneficial action.

3. Observations indicated that the effectiveness of any fretting-corrosion inhibitor is dependent on its ability to prevent metallic adhesion continuously.

Lewis Flight Propulsion Laboratory,  
National Advisory Committee for Aeronautics,  
Cleveland, Ohio, June 23, 1950.

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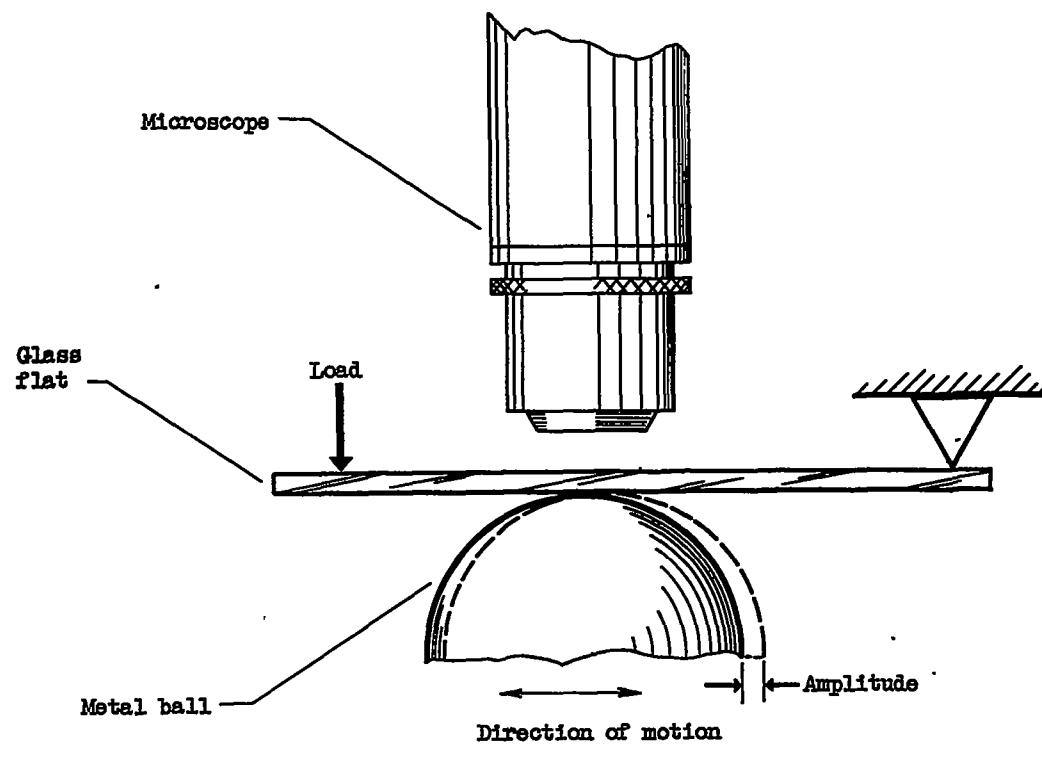
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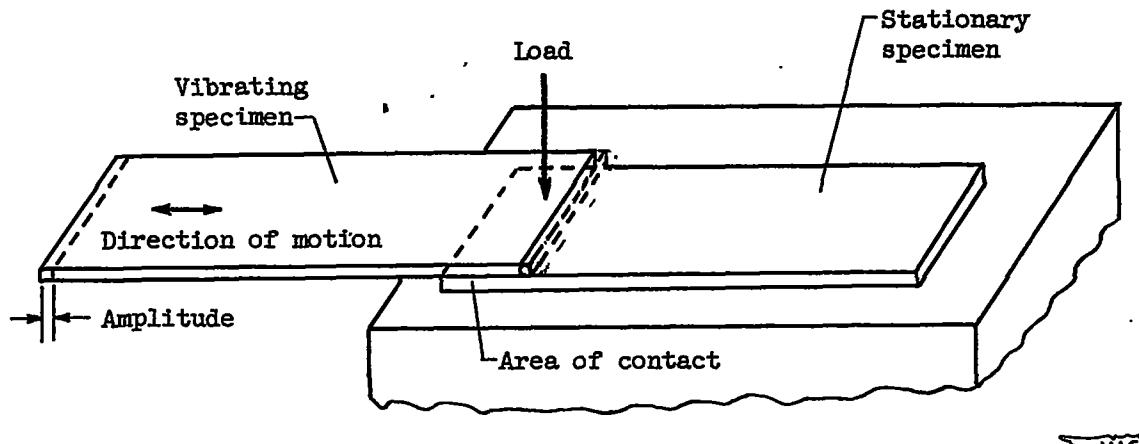
TABLE I - RESULTS OF FRETTING-CORROSION INVESTIGATION  
USING MOLYBDENUM DISULFIDE  $\text{MoS}_2$  AS INHIBITOR

Method	Surface condition	Average number of cycles for first evidence of fretting corrosion	Observation of contact area during and after experiments
Steel ball against glass flat			
-	Clean	1-30	Instantaneous fretting corrosion
1	$\text{MoS}_2$ dusted	72,000	$\text{MoS}_2$ formed smooth bearing surface then rubbed away
2	$\text{MoS}_2$ rubbed	21,600	$\text{MoS}_2$ smeared thinly then rubbed away
3	$\text{MoS}_2$ , water, and aerosol	21,600	$\text{MoS}_2$ visible only in surrounding area
4	$\text{MoS}_2$ and oil	86,400	$\text{MoS}_2$ visible only in surrounding area
5	$\text{MoS}_2$ and grease	216,000	$\text{MoS}_2$ visible only in surrounding area
6	Bonded $\text{MoS}_2$	28,000,000	Coating formed smooth bearing surface then brown stain appeared
Steel flat against steel flat			
-	Clean	< 100	Immediate fretting corrosion
1	$\text{MoS}_2$ dusted	100,000-160,000	Dry red oxide and stain
4	$\text{MoS}_2$ and oil	700,000-760,000	Brown-black debris and spotty surface failure and stain
5	$\text{MoS}_2$ and grease	1,560,000-1,620,000	Brown-black debris and spotty surface failure and red stain
6	Bonded $\text{MoS}_2$	9,823,000-9,883,000	Smooth shiny bearing areas formed then brown powder appeared





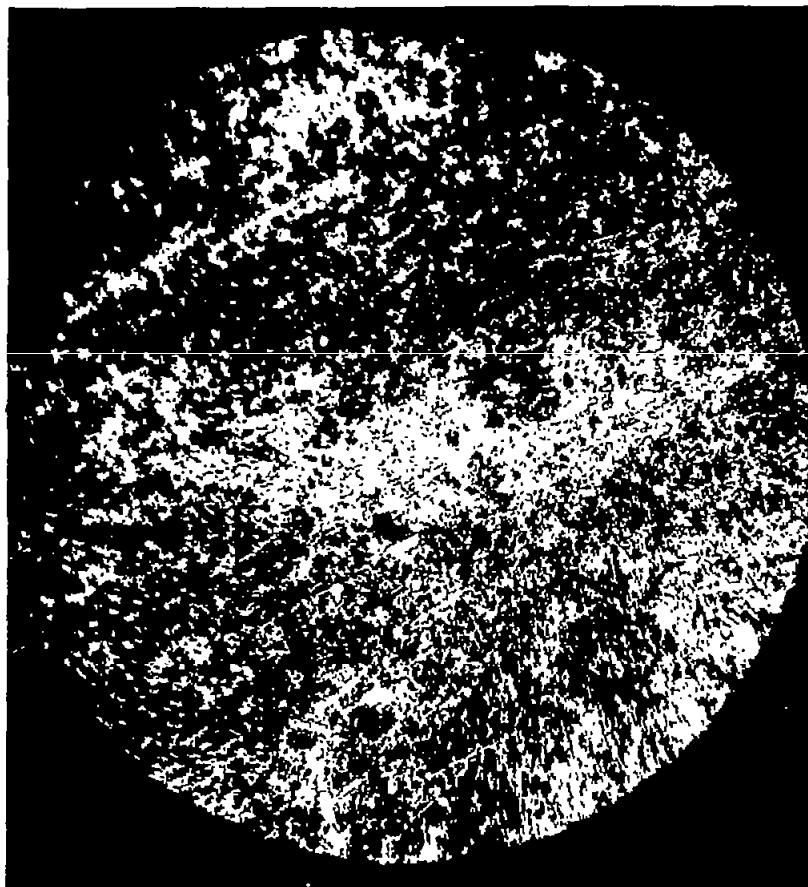
(a) Steel ball against glass flat.



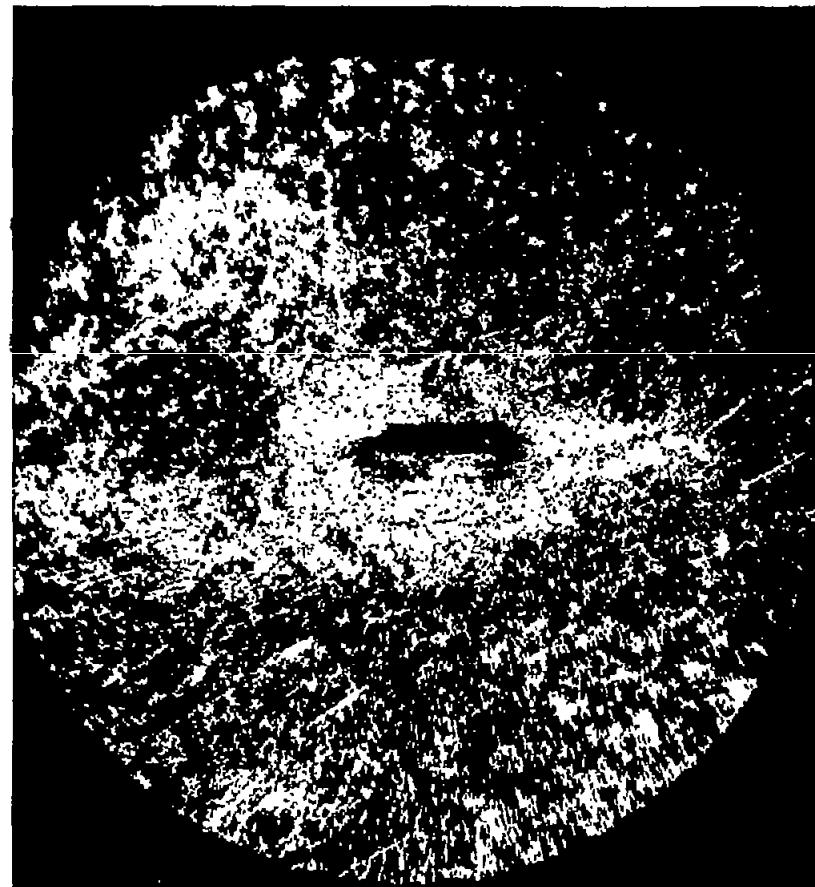
(b) Steel flat against steel flat.

Figure 1. - Schematic diagram of fretting-corrosion apparatus.





(a) Before fretting corrosion, 0 cycles.

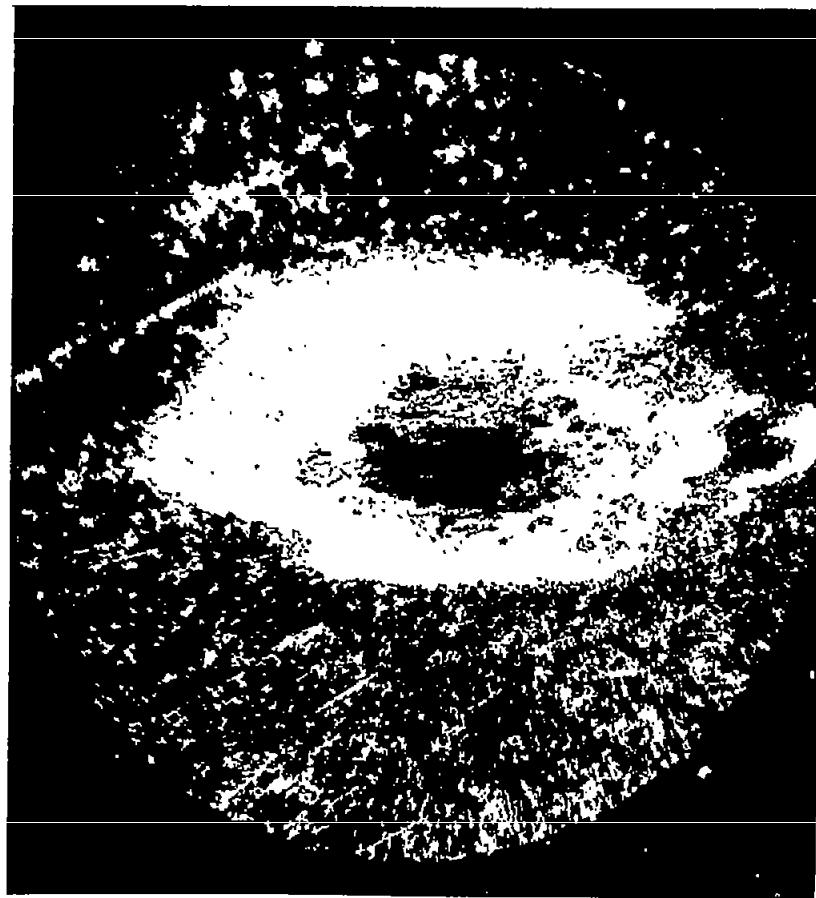


(b) After fretting corrosion, 30 cycles.

Figure 2. - Photomicrographs of specimens before and after fretting corrosion caused by vibrating clean chrome-alloy steel ball in contact with glass microscope slide. X140.

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(c) After fretting corrosion, 120 cycles.

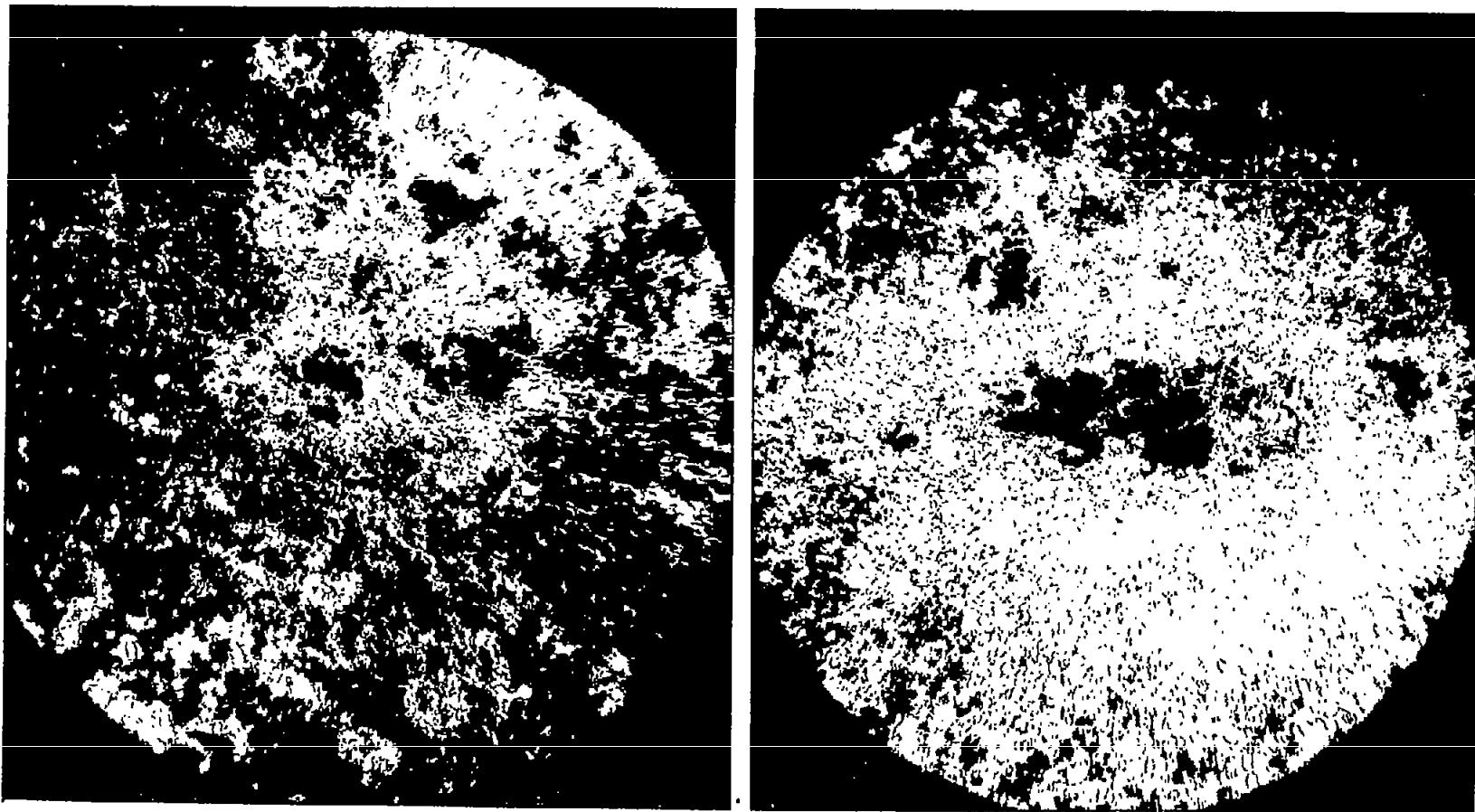


(d) After fretting corrosion, 360 cycles.

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Figure 2. - Concluded. Photomicrographs of specimens before and after fretting corrosion caused by vibrating clean chrome-alloy steel ball in contact with glass microscope slide, X140.





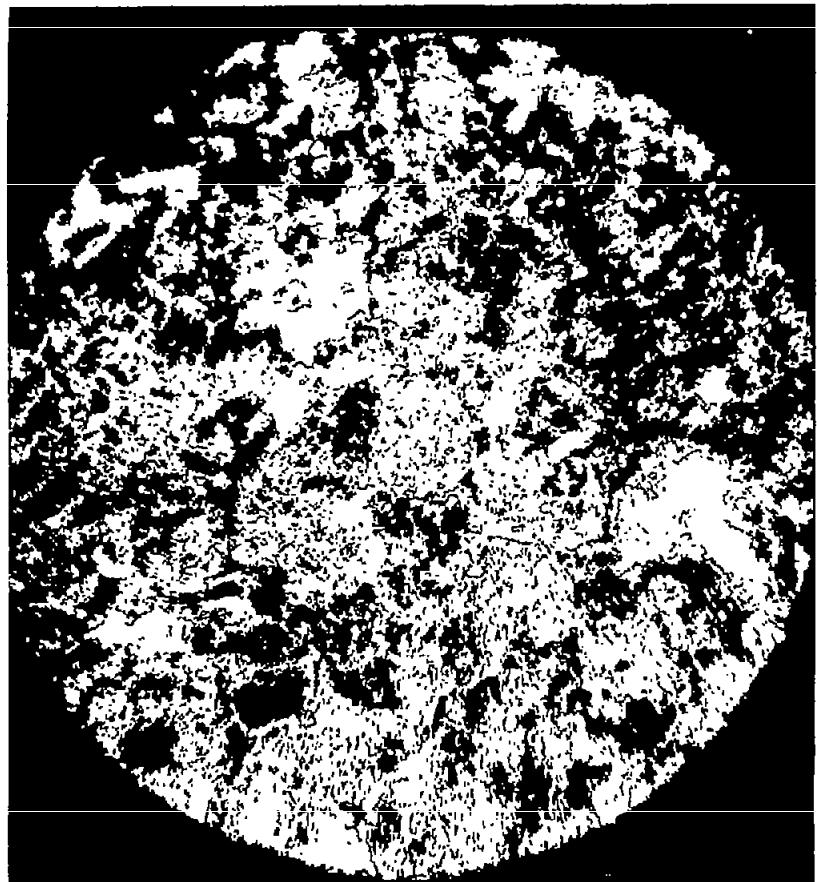
(a) Before fretting corrosion, 0 cycles.

(b) After fretting corrosion, 72,000 cycles.

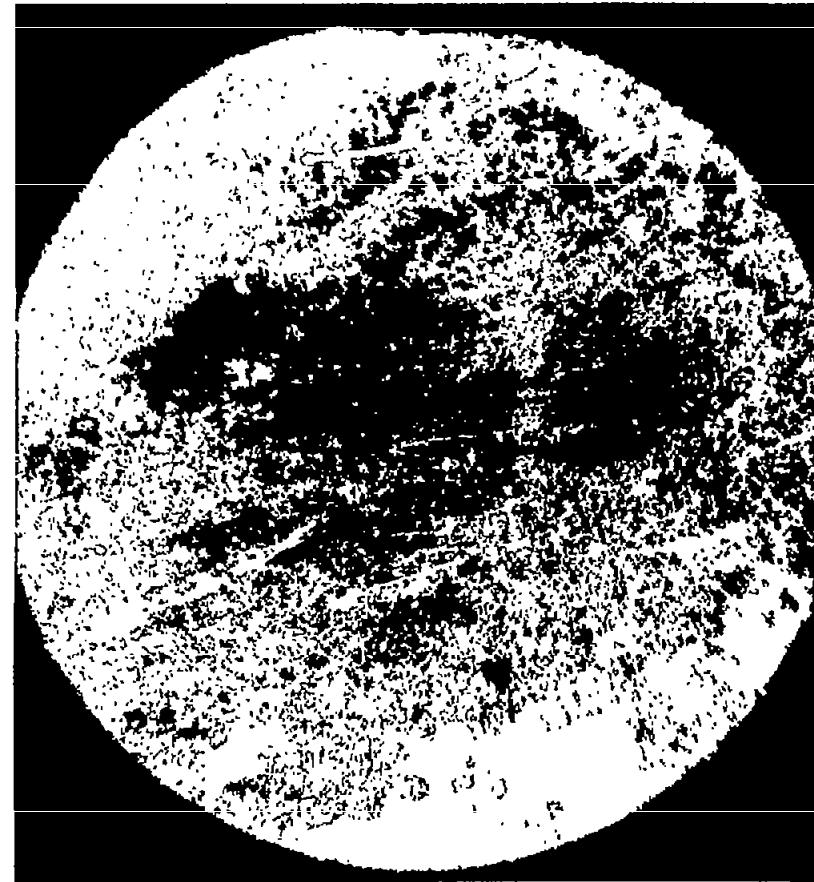
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Figure 3. - Photomicrographs of specimens before and after fretting corrosion caused by vibrating chrome-alloy steel ball dusted with molybdenum disulfide powder in contact with glass microscope slide. X140.





(a) Before fretting corrosion, 0 cycles.

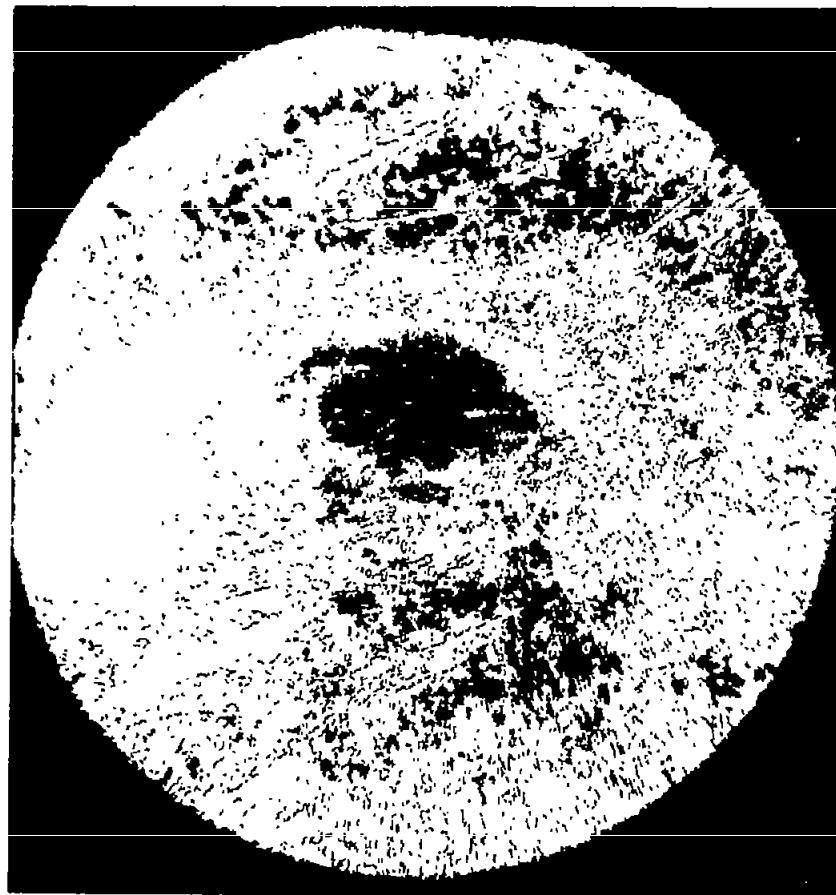


(b) Before fretting corrosion, 360 cycles.

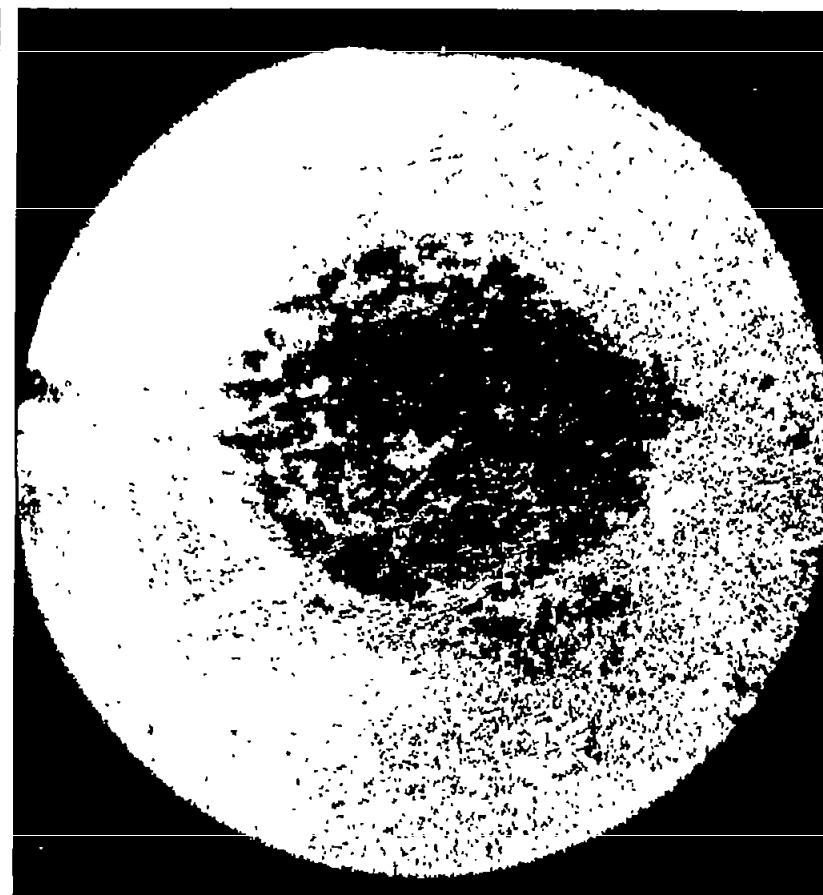
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Figure 4. - Photomicrographs of specimens before and after fretting corrosion caused by vibrating chrome-alloy steel ball immersed in mixture of molybdenum disulfide and light oil in contact with glass microscope slide. X140.





(c) After fretting corrosion, 86,400 cycles.

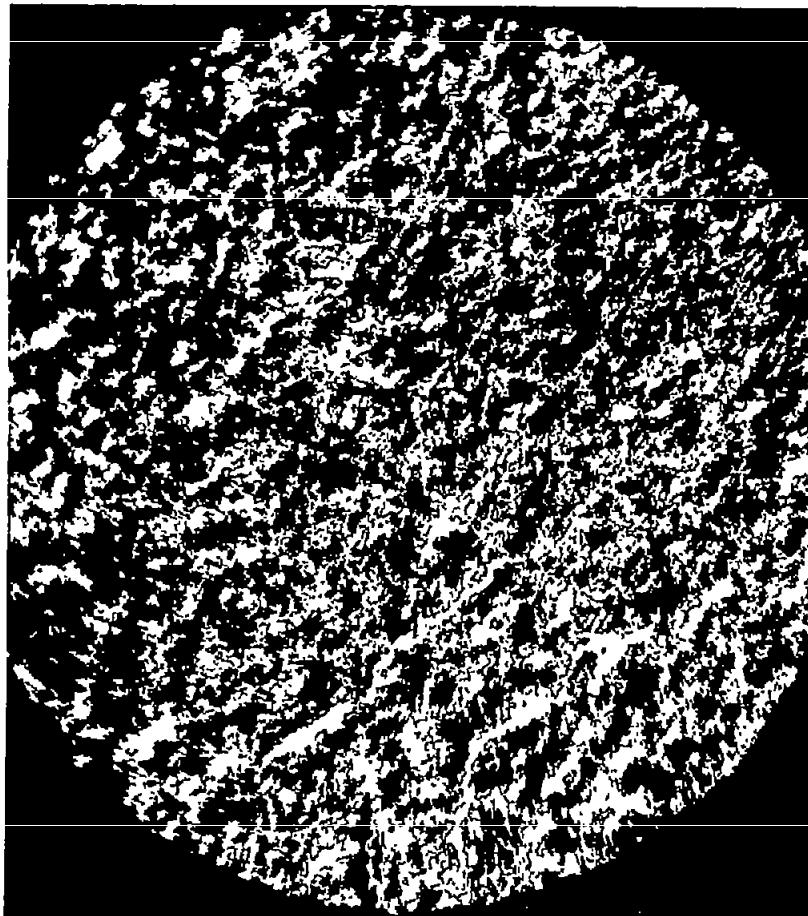


(d) After fretting corrosion, 175,000 cycles.

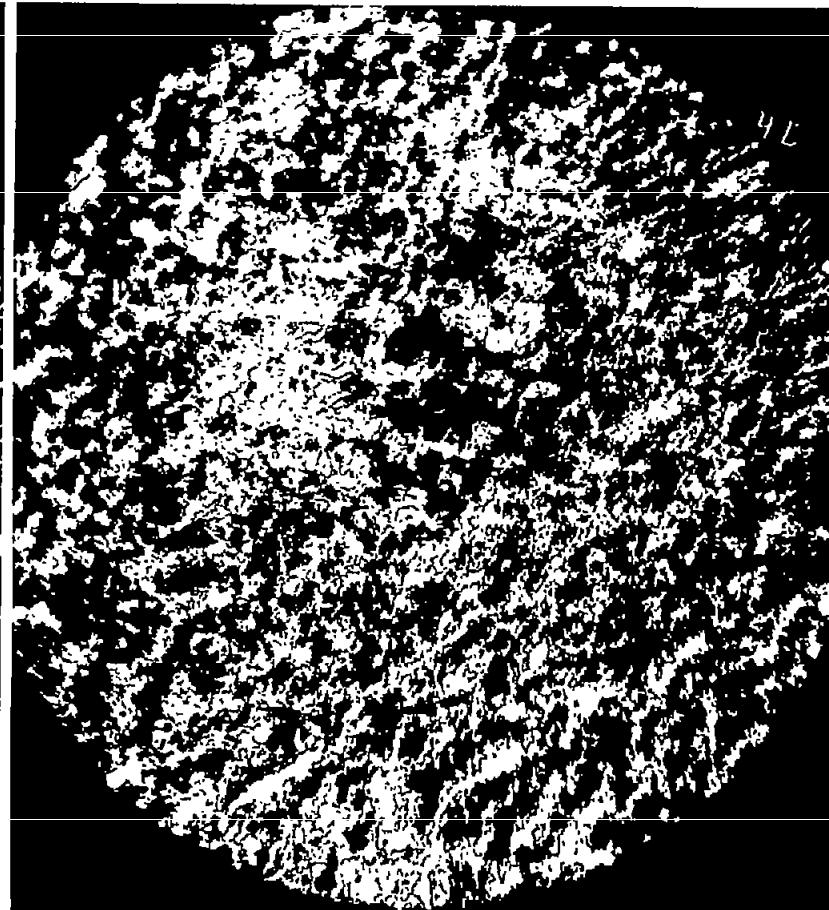
Figure 4. - Concluded. Photomicrographs of specimens before and after fretting corrosion caused by vibrating chrome-alloy steel ball immersed in mixture of molybdenum disulfide and light oil in contact with glass microscope slide. X140.

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(a) Before fretting corrosion, 0 cycles.

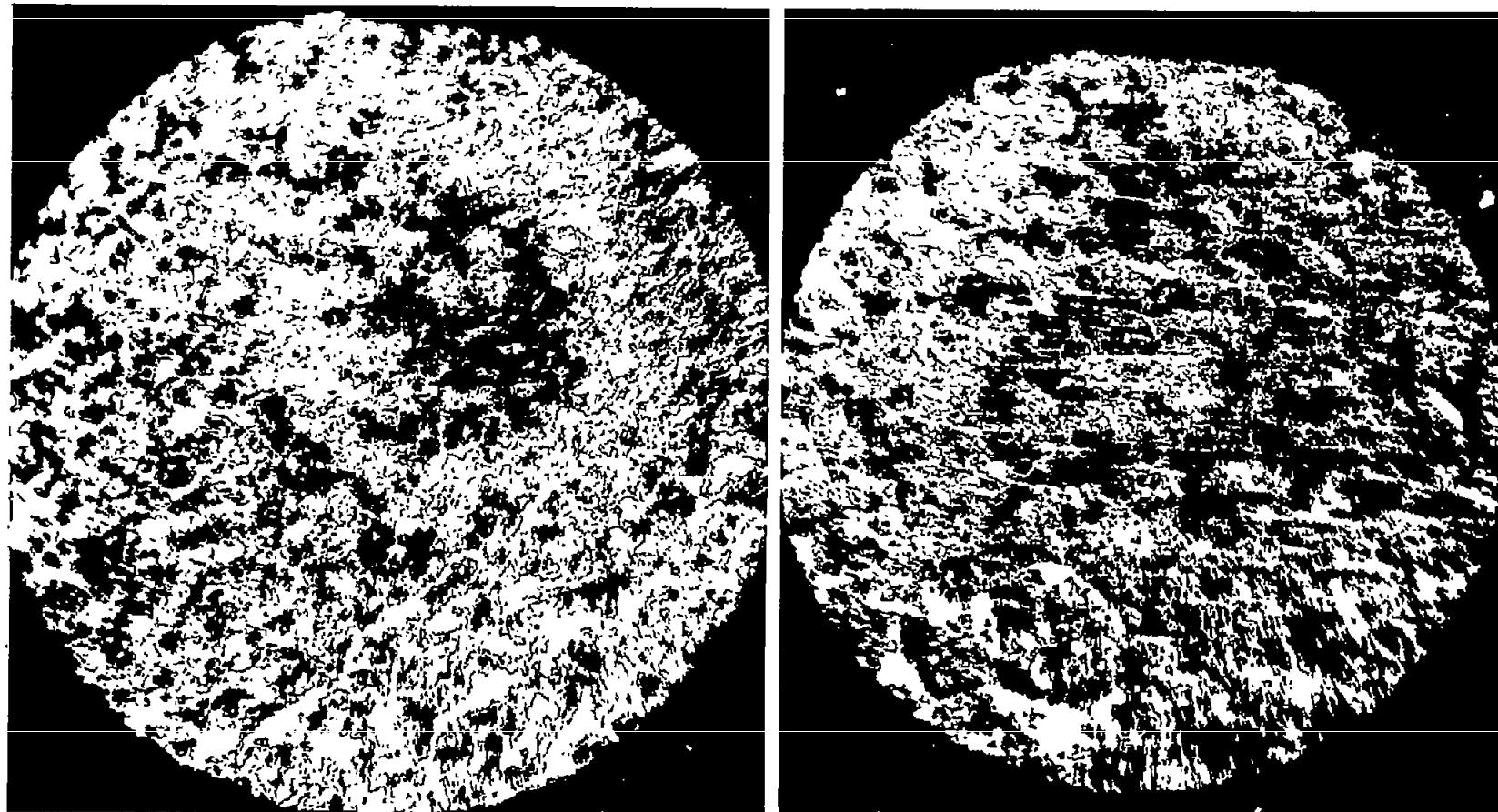


(b) Before fretting corrosion, 144,000 cycles.

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Figure 5. - Photomicrographs of specimens before and after fretting corrosion caused by vibrating chrome-alloy steel ball coated with bonded molybdenum disulfide in contact with glass microscope slide. X140.





(a) Before fretting corrosion, 500,000 cycles.

(d) After fretting corrosion, 28,000,000 cycles;  
without glass.

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Figure 5. - Concluded. Photomicrographs of specimens before and after fretting corrosion caused by vibrating chrome-alloy steel ball coated with bonded molybdenum disulfide in contact with glass microscope slide. X140.



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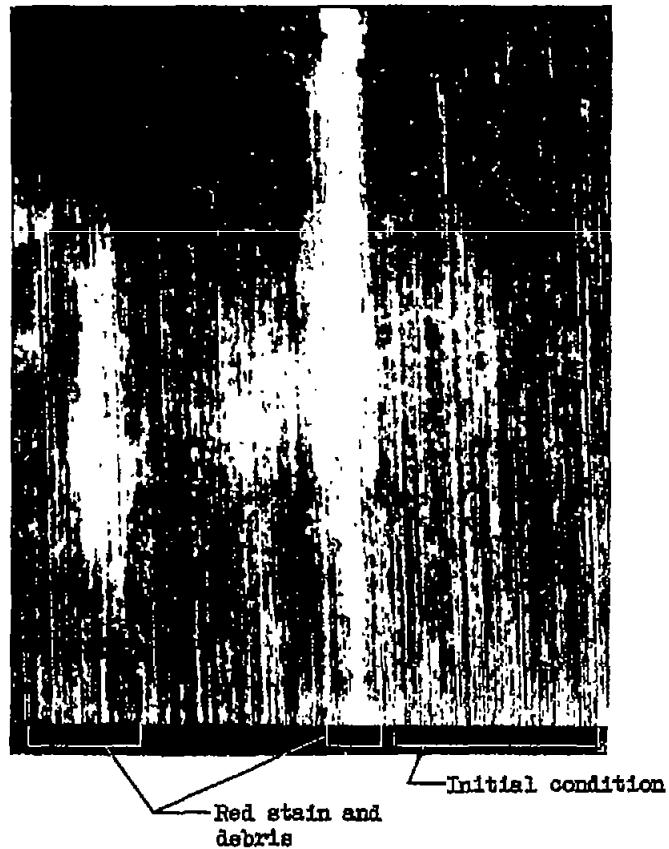


Figure 6. - Macrograph showing fretting corrosion caused by vibrating two contacting flat specimens of clean blue Swedish spring steel for 4500 cycles. X5.

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Figure 7. - Macrograph showing fretting corrosion caused by vibrating blue Swedish spring steel coated with mixture of molybdenum disulfide and heavy grease in contact with clean steel for 1,620,000 cycles. X5.

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